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IS 3134 (1992): Menthol [PCD 18: Natural and Synthetic Fragrance Materials]

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भारतीय मानक

मेंथोल — विशिष्टि

(पहला पुनरीक्षण)

Indian Standard

MENTHOL — SPECIFICATION

(First Revision)

UDC 668·532·42

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

October 1992

Price Group 2

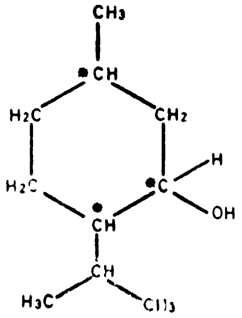
Natural and Synthetic Perfumery Materials Sectional Committee, PCD 18

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Natural and Synthetic Perfumery Materials Secrional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

This standard was first published in 1965. It is being revised with a view to bring it in line with the trade practices prevalent in perfumery industry and also to align it with the quality level of material currently being produced and sold in the country.

Menthol ($C_{10}H_{20}O$) contains three asymmetric carbon atoms* (*see* formula given below). This cyclic, saturated, secondary alcohol may, therefore, exist in four externally compensated and eight optically active forms:



Menthol

(l-Methyl-4- isopropylcyclohexane-3-ol, Molecular mass 156.27)

This standard, however, deals with only, laevo-rotatory l-menthol occurring naturally or produced synthetically. In the earlier version, racemic dl-menthol, produced synthetically, was also covered which is no longer commercially available, hence has been deleted in this revision.

Menthol occurs in the Japanese type oil of mint distilled from *Mentha arvensis.* L which contains 65-70 percent l-Menthol.

In India a phenomenal development for the production of l-Menthol from oil of *Mentha arvensis* and its improved varieties like MAS-1 and *SHIVALIK* has taken place during the period 1970-1990. Presently, *SHIVALIK oil* is the main source for the production of l-menthol. *SHIVALIK mentha* oil contain 75-78 percent l-menthol. l-Menthol is usually obtained by fractional cooling of the mentha oils from + 15°C to − 40°C and subsequently separating the cake of (-) -menthol by centrifuging. Large and bold crystals of l-menthol are then obtained by controlled slow cooling crystallization.

l-Menthol is also manufactured synthetically in India from delta-3-carene. l-menthol can also be prepared from d-citronella ex-citronella oil, menthones and similar other sources.

l-Menthol produced from natural sources is available in the form of powder, flakes, small crystals and big crystals. The minimum purity of powder grade menthol is 98·5 percent whereas that of big crystals is as high as 100 percent.

(*Continued on third cover*)

Indian Standard

MENTHOL — SPECIFICATION

(First Revision)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for menthol largely used for flavoring in candies, pan masala and chewing gums; in compounding of pharmaceutical preparations; in flavoring of tooth pastes, mouth washes, oral preparations; in the preparation of mentholized cigarettes, etc.

2 REFERENCES

The following Indian standards are necessary adjuncts to this standard:

IS No.	*Title*
326	Methods of sampling and test for natural and synthetic perfumery materials
(Part 1) : 1980	Sampling (*second revision*)
(Part 2) : 1980	Preliminary examination of perfumery materials and samples (*second revision*)
(Part 4) : 1980	Determination of optical rotation (*second revision*)
(Part 6) : 1986	Determination of solubility (*second revision*)
(Part 16) : 1989	Determination of melting point (*second revision*)
1070 : 1992	Reagent grade water — Specification (*third revision*)
2284 : 1988	Method of olfactory assessment of natural and synthetic perfumery materials (*first revision*)
6597 : 1988	Glossary of terms relating to natural and synthetic perfumery materials (*first revision*)

The above mentioned standards contain provisions which, through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated above.

3 TERMINOLOGY

For the purpose of this standard, the definitions given in IS 6597 : 1988 shall apply.

4 REQUIREMENTS

4.1 Description

The material shall consist of colourless, acicular or prismatic crystals with a penetrating peppermint like odour. When placed on the tongue the material shall impart a biting sensation and a slightly bitter taste followed by a pleasant feeling of cold.

The material shall also be tested olfactorily and especially for by-notes as prescribed in IS 2284 : 1988.

4.2 Identification Tests

4.2.1 A five percent (*m*/*v*) solution of the material in ethyl alcohol (90 percent by volume) shall be natural to litmus solution.

4.2.2 Dissolve 10 mg of the material in 1 ml of sulphuric acid and add 1 ml of a solution of vanillin in sulphuric acid. An orange-yellow colour shall be produced which, on addition of 1 ml of water, shall further change to violet colour.

4.3 Freedom from Thymol and Other Phenols

Dissolve a few crystals of the material in 1 ml of glacial acetic acid, add three drops of sulphuric acid and one drop of nitric acid. No green colour shall develop.

4.3.1 No colour due to the presence of phenolic substances shall be produced when a solution of the material in ethyl alcohol (90 percent by volume) is added to an aqueous solution of ferric chloride.

4.4 Solubility

The material shall be soluble in all proportions of ethyl alcohol (90 percent by volume) when tested as prescribed in IS 326 (Part 6) : 1986.

4.5 The material shall also comply with the requirements of **Table 1**.

Table 1 Requirements for Menthol

(*Clause* 4.5)

Sl No.	Characteristic	Requirement	Method of Test, Ref to
(1)	(2)	(3)	(4)
i)	Colour and appearance	Colourless prismatic crystals	IS 326 (Part 2) : 1980
ii)	Melting range, °C	42 - 44	IS 326 (Part 16) : 1989
iii)	Optical rotation (10 percent (*m/v*) solution in 95 percent ethyl alcohol)	- 49° to - 50°	IS 326 (Part 4) : 1980
iv)	Non-volatile matter, percent by GC, *Max*	0.05	Annex A
v)	Menthol content, percent by GC, *Min*	99	Annex B

5 PACKING AND MARKING

5.1 Packing

The material shall be supplied in air tight well-closed containers permitting a minimum of air space, as agreed to between the purchaser and the supplier.

5.1.1 The material shall be well-protected from light and stored in a cool place.

5.2 Marking

The material shall be marked with the following information:

a) Name of the material;

b) Each container shall be clearly marked with the origin of the material and the menthol content (total alcohols);

c) Indication of source of manufacture;

d) Net mass of material; and

e) Batch number.

6 SAMPLING

Representative samples of the material shall be drawn as prescribed in IS 326 (Part 1) : 1984.

7 TESTS

7.1 Tests shall be conducted as prescribed in **4.1** to **4.4** and in col 4 of Table 1.

7.2 Quality of Reagents

Unless otherwise specified, pure chemicals and distilled water (*see* IS 1070 : 1992) shall be employed in tests.

ANNEX A

[(*Table* 1, *Item* iv)]

DETERMINATION OF NON-VOLATILE MATTER

A-1 APPARATUS

Platinum or Silica Dish — about 25 mm in diameter.

A-2 PROCEDURE

Place 5 g of the material in an accurately weighted platinum or silica dish, and place it on a water-bath. Continue heating till most of the material has volatilized. Transfer the dish to a hot air-oven maintained at 105 ± 1°C and heat to constant mass. Cool in a desiccator and weigh.

A-2.1 Calculation

$$\text{Non-volatile matter, percent by mass} = \frac{a \times 100}{b}$$

where

a = mass in g of the residue, and

b = mass in g of the material taken for the test.

ANNEX B

[*Table 1, Item* (v)]

GAS CHROMATOGRAPHIC ANALYSIS OF MENTHOL

B-0 GENERAL

The chromatographic conditions given here are for guidance only.

B-0.1 Outline of the Method

A sample of the material is dissolved in a suitable solvent (for example, hexane, cyclohexane or petroleum ether) and is injected into the gas chromatograph where it is carried by the carrier gas from one end of the column to the other. During its movement, the constituents of the sample undergo distribution at different rates and ultimately get separated from one another. The separated constituents emerge from the end of the column one after another and are detected by suitable means whose response is related to the amount of a specific component leaving the column.

B-1 APPARATUS

B-1.1 Any suitable gas chromatograph and column capable of being operated under conditions suitable for resolving the individual constituents into distinct peaks may be used. The typical chromatogram for menthol with the following chromatographic conditions is shown in Fig. 1.

Sample	**Menthol**
Column	
Material	Stainless steel
Length	12 m
Orifice	0.32 cm
Stationary phase and solid support	FFAP* 10 percent by mass on Chromosorb WHP, 100-120 mesh
Carrier gas	**Nitrogen**
Conditions	
Column temperature	150°C
Injection port temperature	210°C
Detector	
Type	F.I.D
Temperature	230°C

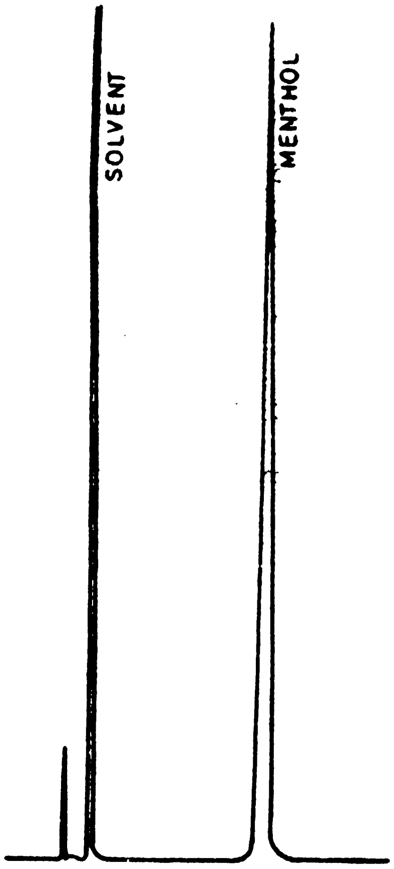


FIG. 1 A TYPICAL CHROMATOGRAM FOR MENTHOL

B-2 CALCULATION

B-2.1 Area Measurement (*See* Note)

Since normal peaks approximate a triangle, the area is measured by multiplying the peak height times the width of half height. The normal peak base is not taken since large deviations may be observed due to tailing or adsorption. This technique is rapid, simple and fairly accurate when peaks are symmetrical and of reasonable width.

NOTE — Other methods of area measurements, namely, triangulation, disc integrator and electronic digital integrator if fixed with GC machine, would be of great advantage.

B-2.2 Area Normalization

By normalizing, it is meant, calculating the percentage composition by measuring the area of each and dividing the individual areas by total area, for example:

$$\text{Percentage of A} = \frac{\text{Area of } A}{\text{Total area}} \times 100$$

---

* Free fatty acid phase (FFAP) in carbowax 20 M treated with nitrophthalic acid.

AMENDMENT NO. 1 JANUARY 1999
TO
IS 3134 : 1992 MENTHOL — SPECIFICATION

(First Revision)

(*Page* 3, *clause* B-1.1, *line* 10)— Substitute '3 m' *for* '12 m'.

(*Page* 3, *clause* B-2) — Delete.

(PCD 18)

Reprography Unit, BIS, New Delhi, India.

(Continued from second cover)

In this revision, a new requirement of purity of menthol content by GC has been included and requirement for melting range has been revised based on data generated through indigenous testing. Requirement for congealing point has been deleted.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analyis, shall be rounded off in accordance with IS 2 : 1969 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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